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Indian Standard

SPECIFICATION FOR SODIUM OLEOSTEARATE, TECHNICAL (SOAP NOODLES)

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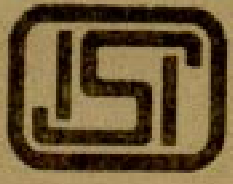

INDIAN STANDARDS INSTITUTION
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
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SPECIFICATION FOR SODIUM OLEOSTEARATE, TECHNICAL (SOAP NOODLES)

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Indian Standard

SPECIFICATION FOR SODIUM OLEOSTEARATE, TECHNICAL (SOAP NOODLES)

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 31 March 1983, after the draft finalized by the Soaps and Other Surface Active Agents Sectional Committee had been approved by the Chemical Division Council.

0.2 When soap is made from conventional raw materials, the saponification of the oil is a relatively simple step and is often combined with the subsequent steps of finishing to a marketable product. The finishing step converts the saponified mass, that is, sodium salt of fatty acids to a form which is ready for consumer use, by suitable processing like milling, plodding, etc, after the incorporation of adjuncts like colour, optical brighteners, perfumes, etc.

0.3 With the need for using non-conventional oils due to shortage of traditional soap making oils and also for the subsequent gylcerine recovery, the soap making process has become involved and capital intensive. Therefore, there is a trend to carry out the capital intensive glycerine recovery operation in one plant and market the intermediate saponified mass for finishing into soap of consumer acceptability to a different establishment.

0.4 Sodium oleostearate, technical (stabilized) contains sodium salt of fatty acids comprising chiefly C-18 unsaturated and saturated acids with added preservatives and necessary electrolytes, built or unbuilt and in a form which is not suitable for direct domestic use, such as soap noodles.

0.5 This standard contains clauses **4.2** and **5.1** which call for agreement between the purchaser and the supplier.

0.5 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960*. The number of significant places retained

*Rules for rounding off numerical values (*revised*).

in the rounded off value should be the same as that of the specified value in this standard.

1. SCOPE

1.1 This standard covers requirements for sodium oleostearate, technical, (soap noodles) used as an intermediate product for subsequent conversion into a marketable soap.

2. TERMINOLOGY

2.1 For the purpose of this standard, the definitions given in **2** of IS : 286 - 1978* and IS : 7597 - 1974† shall apply.

3. TYPES

3.1 Sodium oleostearate is an unfinished soap. It may be classified into the following 3 types:

a) *Type 1* — For toilet soaps,

b) *Type 2* — For pure laundry soaps, and

c) *Type 3* — For built soaps.

4. REQUIREMENTS

4.1 Ingredients — All the ingredients used shall be non-injurious to health and the optical brightening agents, if used, shall be biologically safe.

4.2 The material shall be of acceptable colour, as agreed to between the purchaser and the supplier. It shall be in the form of chips or powder or flakes.

4.3 Odour and Lathering Properties — The material shall not have any fishy or any other disagreeable odour and shall possess good lathering and cleaning properties.

4.4 The material shall also comply with the requirements given in Table 1.

5. PACKING

5.1 The material shall be packed as agreed to between the purchaser and the supplier.

*Methods of sampling and test for soaps (*second revision*).

†Glossary of terms relating to surface active agents.

TABLE 1 REQUIREMENTS FOR SODIUM OLEOSTEARATE, TECHNICAL (SOAP NOODLES)

(*Clauses* 4.4, 8.1 *and* 8.3)

Sl No	Characteristic	Requirement For			Method of Test, Ref to	
		Type 1	Type 2	Type 3	Appendix in This Standard	Cl No. in IS : 286-1978*
(1)	(2)	(3)	(4)	(5)	(6)	(7)
i)	Total fatty matter, percent by mass, *Min*	76·0	62·0	45·0	—	15
ii)	Free caustic alkali as sodium hydroxide (NaOH), percent by mass, *Max*	0·05	0·1	0·2	—	6·2
iii)	Matter insoluble in alcohol, percent by mass, *Max*	2·5	2·5	20·0	—	5
iv)	Titre of total fatty acids, °C, *Min*	37	33	33	—	16
v)	Chlorides (as sodium chloride), percent by mass, *Max*	1·5	2·0	3·0	—	10
vi)	Free carbonated alkali, percent by mass, *Max*	1·0	1·0	1·0	—	28
vii)	Nickel content (as Ni), parts per million, *Max*	0·2	—	—	A	—
viii)	Iron (as Fe) content, parts per million, *Max*	30	—	—	B	—
ix)	Copper (as Cu) content, parts per million, *Max*	3	—	—	C	—

*Methods of sampling and test for soaps (*second revision*).

6. MARKING

6.1 The packages shall be securely closed and marked with the following particulars:

a) Name of the manufacturer;

b) Name of material, type and its recognized trade-mark, if any;

c) Batch number or lot number in code or otherwise; and

d) Month and year of manufacture.

6.1.1 The packages may also be marked with the ISI Certification Mark.

NOTE — The use of the ISI Certification Mark is governed by the provisions of the Indian Standards Institution (Certification Marks) Act and the Rules and Regulations made thereunder. The ISI Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well-defined system of inspection, testing and quality control which is devised and supervised by ISI and operated by the producer. ISI marked products are also continuously checked by ISI for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the ISI Certification Mark may be granted to manufacturers or processors, may be obtained from the Indian Standards Institution.

7. SAMPLING

7.1 For this purpose, general precautions, scale of sampling and preparation of test samples shall be as prescribed in **3.1** and **3.2** of IS : 286 - 1978* respectively.

8. TESTS

8.1 Tests for the determination of characteristics given at Sl No. (i) and (ii) in Table 1 shall be conducted on each of the individual samples separately.

8.2 Tests for determination of the remaining characteristics shall be conducted on composite sample.

8.3 Tests to evaluate the characteristics specified in Table 1 shall be conducted as prescribed in IS : 286 - 1978*, Appendices A, B and C. References to the relevant clauses of IS : 286 - 1978* and Appendices A to C, are given in col 6 and 7 of Table 1.

8.4 Quality of Reagents — Unless specified otherwise, pure chemicals and distilled water (*see* IS : 1070 - 1977†) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of the analysis.

APPENDIX A

[*Table* 1, (*Item* vii)]

DETERMINATION OF NICKEL

A-0. OUTLINE OF THE METHOD

A-0.1 The method is based on the isolation of metal from the soap and reaction between nickel in the oxidised form with dimethyl glyoxime

*Methods of sampling and test for soaps (*second revision*).

†Specification for water for general laboratory use (*second revision*).

forming a red colour, the intensity of which is proportional to the amount of nickel present in the sample.

A-1. APPARATUS

A-1.1 UV Spectrophotometer

A-2. REAGENTS

A-2.1 Sodium Hydroxide — Solid.

A-2.2 Concentrated Hydrochloric Acid — *See* IS : 265-1976*.

A-2.3 Dimethyl Glyoxime Solution — 0·1 percent (*v/v*) in 95 percent ethyl alcohol.

A-2.4 Saturated Bromine Water

A-2.5 Standard Nickel Sulphate Solution — Containing 1 000 µ g of nickel (Ni) per ml.

A-2.6 Liquor Ammonia — Relative density 0·9.

A-3. PROCEDURE

A-3.1 Isolation of Metals from Sodium Oleostearate — Weigh 50 g of the sample in a beaker and dissolve in hot water. Add to this soap solution 40 ml of concentrated hydrochloric acid, stir and keep on steam bath until fatty acid layer separates. Add 20 g of paraffin wax; stir at intervals, and allow it to settle until phases are clear. Cool to room temperature.

A-3.1.1 Remove wax cake with rod; rinse with water; add rinsings to aqueous phase. Evaporate aqueous phase to about 60 ml by gentle boiling. Add 100 ml of water and filter through paper washed previously with hydrochloric acid. Evaporate the filtrate and washings to about 60 ml. Cool and transfer to volumetric flask. Make up the volume to about 100 ml. Take aliquot portions for metal estimation.

A-3.2 Determination of Nickel Content — Take 50 ml aliquot of the aqueous solution from the test solution in a 250-ml breaker. Evaporate the solution to about 15 ml by heating. Transfer the solution to a 50-ml glass stoppered volumetric flask using a small quantity of water for rinsing the solution from the beaker into the volumetric flask. Add to the flask 3 ml of saturated bromine water and allow to stand for one minute. Add liquor ammonia dropwise until excess bromine is destroyed as

*Specification for hydrochloric acid (*second revision*).

indicated by the disappearance of brown colour. Then add 5 ml of liquor ammonia in excess. If a precipitation occurs, filter the solution and wash the precipitate with water, combine the filtrate and the washings and concentrate to a volume of a few millilitre and transfer to a 50-ml volumetric flask. Add 10 ml of dimethyl glyoxime solution, followed by 15-20 ml of 95 percent ethyl alcohol. Mix thoroughly and make up the volume and again mix thoroughly. Allow the solution to stand for 5 minutes to permit full development of colour and take the absorption/transmittance reading at 445 nm.

Prepare and conduct blank determination simultaneously and similar in all respects. The transmittance of the blank should be 98 ± 1 percent. Determine the nickel content of the sample by reference to a concentration-transmittance graph prepared as follows.

A-3.3 Preparation of Concentration-Transmittance Graph — Weigh accurately 2·261 7 g of nickel sulphate (99 percent $NiSO_4 \cdot 6H_2O$) and dissolve in distilled water in a 500-ml volumetric flask. Add 30 ml of concentrated hydrochloric acid and bring to volume. This solution contains 1 000 µg of nickel per ml. Make appropriate dilutions of this solution and process this solution as in sample above. The dilutions should cover a range 0-100 µg. Finally plot a curve relating transmittance to micrograms of nickel.

A-3.4 Calculation

$$\text{Nickel content, parts per million} = \frac{M_1 - M_2}{m}$$

where

M_1 = micrograms of nickel present in sample,
M_2 = micrograms of nickel present in blank, and
m = mass in gram of the sample taken for the test.

APPENDIX B

[*Table* 1, (*Item* viii)]

DETERMINATION OF IRON

B-0. OUTLINE OF THE METHOD

B-0.1 The method is based on the isolation of metal from the soap by dissolving in hot water. The aqueous extract is treated with citric acid

to sequester aluminium and then thioglycolic acid in ammoniacal solution is added and colour measured spectrophotometrically.

B-1. APPARATUS

B-1.1 Spectrophotometer

B-2. REAGENTS

B-2.1 Liquor Ammonia — Relative density 0·9.

B-2.2 Dilute Sulphuric Acid — 50 percent (*v*/*v*).

B-2.3 Citric Acid (Aqueous Solution) — 50 percent (*v*/*v*).

B-2.4 Thioglycolic Acid (Aqueous Solution)

B-2.5 Standard Iron Solution — Containing 10 μg of iron per ml prepared from ferric ammonium sulphate [$Fe_2(SO_4)_3 . (NH_4)_2 SO_4, 12H_2O$] in acid solution.

B-2.6 Methyl Red Indicator — 0·1 percent aqueous solution.

B-3. PROCEDURE

B-3.1 Isolation of Metals from Sodium Oleostearate — Weigh 50 g of the sample in a beaker and dissolve it with hot water. To this soap solution add 40 ml of concentrated hydrochloric acid with constant stirring and keep the beaker on steam bath until fatty acid layer separates. Add 20 g of paraffin wax to this solution while hot. Stir the solution at intervals and allow it to settle until phases are clear. Cool the mass to room temperature. Remove the wax cake with rod, rinse with water and add the rinsings to the aqueous phase. Evaporate the aqueous phase to about 60 ml by gentle boiling. Add 100 ml of water and filter through paper washed previously with hydrochloric acid. Evaporate the filtrate and washings to about 60 ml. Cool and transfer the solution to a volumetric flask and make up the volume to 100 ml. Take aliquot portion of the solution for metal estimation.

B-3.2 Determination of Iron — Take 5 ml aliquot of the aqueous solution from the test solution in a 25-ml volumetric flask. To this add 4 ml citric acid solution and 0·02 ml methyl red indicator and liquor ammonia till the colour of the solution turns yellow. Then add 3 ml liquor ammonia in excess. Cool the solution and add 3 ml thioglycolic acid. Make up the volume to 25 ml and mix the solution thoroughly. Filter the solution through acid washed and dried filter paper. Measure absorbance of the clear solution at 540 nm in the spectrophotometer

using water as reference. Prepare a calibration curve with standard iron solution and determine the iron content of the soap sample from it.

APPENDIX C

[*Table* 1, (*Item* ix)]

DETERMINATION OF COPPER

C-0. OUTLINE OF THE METHOD

C-0.1 The method is based on the isolation of metal from the soap and to make a copper complex using zinc dibenzyl dithiocarbamate in carbon tetrachloride solution and measure the colour of the solution spectrophotometrically.

C-1. APPARATUS

C-1.1 Spectrophotometer

C-2. REAGENT

C-2.1 Zinc Dibenzyl Dithiocarbamate Solution — 0·05 percent in carbon tetrachloride.

C-2.2 Standard Copper Solution — Containing 1 µg of copper per ml (prepared from a stock solution of 100 times the concentration).

C-3. PROCEDURE

C-3.1 Isolation of Metal from Sodium Oleostearate — Weigh 50 g of the sample in a beaker and dissolve it with hot water. To this soap solution add 40 ml of concentrated hydrochloric acid with constant stirring and keep the beaker on steam bath until fatty acid layer separates. Add 20 g of paraffin wax to this solution while hot. Stir the solution at intervals and allow it to settle until phases are clear. Cool the mass to room temperature. Remove the wax cake with rod, rinse with water and add the rinsings to the aqueous phase. Evaporate the aqueous phase to about 60 ml by gentle boiling. Add 100 ml of water and filter through paper washed previously with hydrochloric acid. Evaporate the filtrate and washings to about 60 ml. Cool and transfer the solution to a volumetric flask and make up the volume to 100 ml. Take aliquot portion of the solution for metal estimation.

C-3.2 Determination of Copper — Take 20 ml aliquot of the aqueous solution and to it add 10 ml of zinc dibenzyl dithiocarbamate solution followed by 25 ml of sulphuric acid in a separating funnel. Shake the solution for one minute and allow it to settle. Run the lower carbon tetrachloride layer into 25-ml volumetric flask. Wash the aqueous layer with carbon tetrachloride and transfer through glasswool to volumetric flask. Make up the volume and mix well. Measure absorption of the clear solution at 435 nm in the spectrophotometer. Prepare a calibration curve with standard copper solution and determine the copper content of the soap sample from the curve.

NOTE — The standard solution shall also be extracted with carbon tetrachloride before estimation of colour.

(*Continued from page* 2)

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